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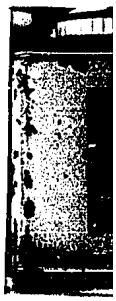

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INVESTIGATION OF METAL POWDERS WITH THE AID OF AN ELECTRON MICROSCOPE

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[Diagrams referred to are appended]

Present methods for studying the grain of metal-ceramic hard alloys (microscopic examination, dispersion analysis, etc.) in most cases give only an approximate characteristic. For example, in an ordinary microscope, determination of grain sizes less than 0.5 micron is impossible.

Other methods of determining graininess and so-called "activity" of powders (methods of oxidation, adsorption, etc.) are comparative, allowing only an indirect estimate of the total specific surface of powders.

The authors have used the electron microscope. In working out the method used, they were assisted by N. G. Sushkin.

Specimens for examination were prepared by two methods: by precipitating the powder from some liquid medium on a previously prepared collodion film, and by preparation of a film with the powder to be studied included in the film.

The latter method is used as follows. A small amount of the powder is poured into an agate mortar, to which is added enough lac to make a paste-like mass upon light mixing, and thicker with further mixing. To this is gradually added a 3% lac solution, thinning the mass to a concentration where a drop of the powder-lac mixture will form a film of the desired thickness.

Introducing large and heavy particles into the film may have a negative effect on the film's strength; fine grain powders maintain the film's strength.

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To increase the strength of the film, experiments were conducted using additives in the lac. Turpentine appeared to be most suitable. In a comparison of the two methods described, the latter method appears preferable, since the preliminary pulverization of the powder with the collodion lacquer to a certain degree facilitates dispersion of the powder and crushing of conglomerates.

As a result of the powder's being included in the film, there is no additional conglomeration upon desiccation. This is, however, possible when the first method is used; in addition, this method is faster and less tedious. However, in the case of easily crushed powders (WO_3, H_2WO_4, etc.), it is faulty due to the fact that there is a difference between the sample taken for analysis and the original powder, this difference being the result of the additional grinding with the lacquers.

To eliminate this fault, the authors used a mechanical vibrator where dispersion of the powder is accomplished with the use of audio-frequency oscillations and further crushing is eliminated. The powders were examined at magnifications of 6,000-10,000. In some cases, during the examination of very finely dispersed powders, further magnification was accomplished by optical methods.

Examination of wolfram trioxide powder showed that it consists of extremely fine crystals, less than 0.5 micron, approaching a true cubic form. The powder of wolframic acid appeared equally as fine grained.

Results of the examination of metallic wolfram powders obtained by reduction from wolfram trioxide are given in Table 1.

Table 1.

Wolfram Powder and Method of Obtaining	Dry Wt (g/cu cm)	Grain Form	Graininess Characteristic
Wolfram No. 6, reduced with hydrogen at 800°	1.25	Hexahedron	Fine grain; basic grain mass approx 0.5 micron; max. size 1.5 microns. There was an extremely fine fraction with a grain size beyond the microscope's resolving capacity.
Wolfram No. 8, reduced with hydrogen at 750°	1.72	Near Hexahedron	Finegrain powder; uniform graininess; basic grain mass 0.8 micron.
Wolfram No. 9, reduced with hydrogen at 830°	-	Rounded	Coarse, nonuniform grain.
Wolfram No. 7, reduced with hydrogen at 1200°	3.24	Nearly hexahedron	Coarse-grained, grain size 1-4 microns.
Wolfram, reduced with carbon (retort reduction)	-	Somewhat rounded	Nonuniform graininess.

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It is seen from these data that the wolfram grain in most cases has a regular form approaching the cubic. A distinguishing property of some of the wolfram powders (Nos. 6 and 9) is the great tendency toward conglomeration and mutual contact of grains. Wolfram No. 6 conglomerated to such an extent that despite the use of various dispersion methods, separate grains of the powder could not be obtained in the microscope's field of view. The wolfram examined was slightly oxidized, as indicated by the presence of peculiar "tendrils", growing on the surface of the wolfram grains and representing, apparently, the lower oxides of wolfram.

The temperature of reduction is noticeably reflected in the graininess of the powders obtained: the higher the temperature, the coarser the powders. The dry weight of the powders coincides well with the character of their graininess. For example, the fine grained powders Nos. 6 and 8 have a low dry weight while that of No. 7 is at a maximum.

Examination of the carbon-reduced wolfram showed that this powder is extraordinarily nonuniform in graininess, which contradicts an earlier opinion concerning its fine grain and uniformity.

On a photomicrograph of the retort wolfram, the presence of a large number of free carbon particles (carbon black) was noted. These particles appeared light gray since they are more permeable to electron beams.

The results of the examination of wolfram carbide (WC) powders obtained under nearly the same conditions of carburization and pulverization from wolfram powders Nos. 6 and 7 are presented in Table 2.

Table 2. Results of the Examination of Wolfram Carbide

Powder and Method of Obtaining	C_{total}	C_{free}	Dry Wt g/cu cm	Wolfram Carbide Characteristic
Wolfram Carbide No. 3	6.17	0.05	1.96	Fine grain; grain of irregular splinter form.
Wolfram Carbide No. 5, prepared from Wolfram No. 7	5.88	0.04	3.09	Coarse-grain powder, nonuniform, basic grain mass, 2 microns: grains are of irregular form with ragged edges.

Investigations showed that the graininess of the carbide powder is related to the graininess of the initial wolfram; viz., the fine grained carbide corresponds to fine grained wolfram. The dry weight of the wolfram carbide increases as coarseness of the powder increases, within the given limits. In all carbide specimens examined, the irregular "sliver" form of the WC grains was notable, this form having been generated during the carburization of the wolfram powder grains.

A series of wolfram and wolfram carbide powders were obtained from ammonium parawolframate. The "parawolframate" carbide is characterized by several properties which distinguish it from the wolfram carbide obtained from ordinary wolfram trioxide. In particular, it develops less of a tendency toward grain growth during the sintering of metal ceramic hard alloys, even with a considerable increase in temperature above that encountered in ordinary technology, and yields an alloy comparatively uniform in graininess -- without accumulations of coarse grains of wolfram carbide.

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Figure 1 shows the form of a grain (crystal) of ammonium parawolframate powder; upon crushing in an agate mortar to break up conglomerates and prepare the specimen for examination in the electron microscope, the parawolframate is highly pulverized into a very fine powder with grain sizes of the order of 0.03-0.6 microns. The basic grain mass is so small that the size of the grains may be determined only with magnifications of the order of 30,000.

The carbide grains are finer as compared with the initial wolfram grains. In contrast to the ordinarily angular grains of a carbide of some other origin, the parawolframate carbide grains have a rounded form. Powders of this carbide are considerably more uniform in grain, and contain practically no fine fraction.

A study was also made of the change in graininess and grain form of wolfram carbide powders, which (change) occurred as a result of their being crushed in ball mills. Powders subjected to dry and wet (alcohol) crushing for periods of 6, 12, 24, 72, and 120 hours were studied. On the basis of the examination, it was possible to conclude that a noticeable breaking down of the carbide grains is observed after 24 hour's grinding as conducted under ordinary industrial conditions. Wet grinding assures more rapid and effective pulverization of the powders.

In addition, in dry-ground powders, even after long crushing, a considerable amount of comparatively coarse "rolled" particles are found.

Determination of the granulometric composition of fine grained powders is naturally of interest since the "grain collection" plays a very important part in the formation of metal-ceramic alloys.

To construct a distribution curve we took a very fine grain powder of No. 8 wolfram, obtained by reduction with hydrogen at low temperature from wolframic acid.

Data from microscopic analysis using a metal microscope were as follows: grain diameter 0.5 micron, 85%; 1 micron, 15%; and also a large quantity of conglomerates.

Figure 2 shows the curve for grain-size distribution of the same wolfram powder, based on measurements made in the electron microscope. Examination of the curve shows that with the aid of the electron microscope one can determine granulometric composition of a powder with extreme accuracy, observe and measure particles of less than 0.5 micron diameter, and compute the quantity of particles belonging to each group. Figure 3 shows a similar distribution curve for No. 8a wolfram carbide powder. Determination of grain size (i.e., measuring the least grain diameter, d, and the largest diameter, D) of these fine grain powders was done by using electron photomicrographs with 3-x supplementary optical enlargement.

In order to cover the largest possible number of particles we worked with a 3000-x magnification, i.e., minimum electron magnification, at the same time increasing the microscope's field of view to maximum. Even at this magnification, however, the quantity of particles in the field of view was insufficient for constructing a curve.

To increase the amount of measurable particles, the authors used a photograph of a portion of the specimen film, measuring 0.07 mm on a side. This area does not fit in the microscope's field of view and, therefore, it was divided into 15 smaller sections. The sections were photographed and each picture was enlarged 3 times by optical methods, so that total magnification

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amounted to 9000. Thus, 1 micron would measure 9 mm on the photographs. The grains were then measured with a millimeter scale accurate to 0.5 mm. For example, 720 grains were measured to plot the curve in Figure 2.

In conclusion, nickel and cobalt powders were studied. The results are presented in Table 3.

Table 3. Results of the Study of Nickel and Cobalt Powders

Powder	Method and Conditions for Obtaining	Oxygen Content	Dry Wt (g/cu cm)	Characteristic
Nickel No. 1	Roasting nickel oxalate in a current of hydrogen	0.79	1.69	Rounded grain with serrated edges.
Nickel No. 3	Nickel carbonyl	-	-	Rounded grain with well-developed characteristic "toothed" surface.
Cobalt No. 1	Reduced from cobalt oxalate in a current of hydrogen	0.45	0.45	Peculiar grain, has elongated "branch", chain-like appearance.

In the case of the grains of nickel and cobalt powders, there is a peculiar, highly-developed "toothed" surface attesting to great "activity" and tendency of the grains to agglomeration. This peculiar grain form came out during preparation of the specimens for examination in the electron miscroscope.

It has thus been demonstrated that electron photomicrography permits determination of the form and sizes of the very fine powders used in the metal ceramics industry. In the case of a number of powders, their characteristic as determined by ordinary methods of investigation is apparent.

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Fig. 1.

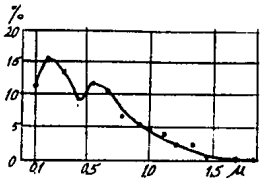

Fig. 2.

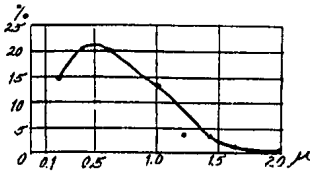

Fig. 3.

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